THE EFFECT OF CADMIUM DOPING ON BSCCO SUPERCONDUCTORS
PREPARED VIA COPRECIPITATION METHOD

By

ALI AGAIL HAMED ABDULGADER

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science

April 2004
DEDICATIONS

To my wife, and my daughters, Fatema and Dwaa
for their love, support and understanding....

To my father, my late mother and family
for their love and concern...
ABSTRACT OF THESIS PRESENTED TO THE SENATE OF UNIVERSITI
PUTRA MALAYSIA IN FULFILMENT
OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE

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Chairman: Professor Abdul Halim bin Shaari, Ph.D.

Faculty: Science And Environmental Studies

The coprecipitation technique was used in the preparation of cadmium doped
Bi_{1.6}Pb_{0.4}Sr_{2}Ca_{2}Cu_{3}O_{10} and Bi_{1.7}Pb_{0.3}Sr_{2}Ca_{2}Cu_{3}O_{10} polycrystalline ceramic
superconductors. In this study, the calcium site was subjected to Cd doping with nominal
composition ranging from x=0 to x=0.1 at different sintering times. The superconducting
properties of the samples have been investigated. The undoped samples, which exhibits
T_{C(R=0)} around 102 –104 K and T_{C(onset)} around 112-116 K, showed large flaky grains
around 9-11 µm in size which are randomly distributed. However, at longer sintering
time (100 h) the undoped sample showed a better orientation as compared to that of the
shorter sintering time (24 hr) sample. The calculated value of Josephson current, I_o,
obtained from the AC susceptibility data showed a much higher value (I_o =100.04 µA)
as compared to the sample prepared by conventional method (I_o =55.9 µA). This
indicates better grain connectivity and higher 2223 phase content, which was confirmed
by SEM photographs. In addition, the nature of the ultra fine particles of the oxalate
powders produced by coprecipitation method have increased the diffusion reaction and shortened the heat treatment procedure for the sample preparation, this leads to better superconducting properties as compared to the samples prepared by conventional solid state technique where its diffusion reaction requires high sintering temperatures for long duration and sometimes several grindings.

The resistivity measurements showed the normal metallic behaviour followed by decreased in $T_{C(R=0)}$ as the cadmium concentration increased due to the decrease in the 2223 phase and an increase in the formation of 2212 phase.

The temperature dependence of ac susceptibility data, $\chi'$, shows the shifting of the onset diamagnetism towards lower temperature as the Cd concentration increased due to the presence of low-$T_C$ phase. The imaginary component, $\chi''$, shows a shift in the intergranular coupling peak, $T_p$, towards lower temperature as the Cd concentration increased. Hence it can be deduced that the dynamic magnetic response of the samples are not only phase dependent but also dependant on the intergranular coupling. The calculated $I_0$ which revealed the quality of the coupling of the grains, showed a decrease in its value as the cadmium concentration increased.

The results of x-ray diffraction (XRD) patterns show that all samples doped with Cd contain 2212 peaks which correspond to the low-superconducting phase. The intensity of these peaks increases towards higher value, as the Cd concentration increases. The volume of 2223 phase decreases gradually as the Cd concentration increases. In
addition, there is a possibility that either Cd$^{2+}$ might have occupied other sites in the sample. When long sintering time was applied, the improvement in superconducting properties was obvious at low doping concentrations $x=0.02$ where the sample was still dominated by 2223 phase. Above that concentration, the grain size decreased and became shorter and thicker, randomly distributed as compared to the undoped samples. It is also observed that the superconducting properties and the microstructure improved when the sample was sintered for 48 hours and 100 hours, the high-$T_C$ phase dominates, indicating that the optimum time must be above 48 hours.

The study shows that the substitution of cadmium in calcium site does not improve the $T_C$ of the BSCCO system. This is due to the formation of low-$T_C$ phase which weakened the coupling of the grains. However, all samples showed an obvious improvement in $T_C$ as the sintering time increases.
ABSTRAK TESIS YANG DIKEMUKAKAN KEPADA SENAT UNIVERSITI
PUTRA MALAYSIA
SEBAGAI MEMENUHI KEPERLUAN UNTUK IJAZAH MASTER SAINS

KESAN PENDOPAN KADMIUM KE ATAS SUPERKONDUKTOR BSCCO
MELALUI KAEDAH PEMENDAKAN BERSAMA

Oleh

ALI AGAIL HAMED ABDULGADER

April 2004

Pengerusi:  Profesor Abdul Halim bin Shaari, Ph.D.

Fakulti:  Sains Dan Pengajian Alam Sekitar

Teknik pemendakan bersama telah digunakan bagi menyediakan superkonduktor seramik polihab lur Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ dan Bi$_{1.7}$Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ yang didopkan dengan cadmium. Dalam kajian yang dijalankan ini, tapak kalsium telah didopkan dengan Cd secara berasingan yang berbeza dengan komposisi nominal di antara julat x=0 hingga x=0.1 pada masa pensinteran yang berbeza. Sifat kesuperkonduksian bagi sampel-sampel ini telah dikaji. Sampel-sampel tulen yang menunjukkan $T_{C(R=0)}$ di sekitar 102-104 K dan $T_{C(onset)}$ di sekitar 112-116 K, menunjukkan butiran yang berkeping-keping yang bersaiz 9-11 µm dan tertabur secara rawak. Walau bagaimanapun, pada suhu persinteran yang panjang (100 j), didapati superkonduktor tulin menunjukkan orientasi yang lebih baik dibandingkan dengan sampel yang diinter pada jangkamasa yang lebih pendek (24 j). Nilai arus Josephson $I_0$, yang diperolehi daripada data AC menunjukkan nilai yang lebih tinggi ($I_0= 100.04 \, \mu A$) jika dibandingkan dengan sampel yang dihasilkan melalui kaedah lazim ($I_0=55.9 \, \mu A$). Ini
menunjukkan sifat sempadan butiran yang baik dan kandungan fasa 2223 yang tinggi dan ini disahkan dengan fotograf SEM. Tambahan pula, partikel yang bersaiz tersangat halus bagi serbuk oksalat yang dihasilkan melalui kaedah pemendakan bersama membantu proses resapan dan memendekkan proses pemanasan dalam penyediaan sampel, menghasilkan sifat superkonduktor yang lebih baik jika dibandingkan dengan sampel yang dihasilkan melalui kaedah tindakbalas pepejal di mana ia memerlukan masa persinteran yang lebih panjang dengan beberapa kali proses pengisaran.

Pengukuran kerintangan menunjukkan sifat logam yang lazim dan diikuti dengan penurunan dalam $T_{C(R=0)}$ apabila peratus pendopan kadmium meningkat disebabkan penurunan dalam fasa 2223 dan peningkatan fasa 2212.

Kebergantungan suhu oleh data ACS, $\chi'$ menunjukkan penganjakan permulaan diamagnet pada suhu rendah yang lebih rendah apabila kepekatan Cd meningkat disebabkan oleh kemunculan fasa rendah $T_C$. Komponen khayalan, $\chi''$, menunjukkan anjakan dalam puncak gandingan antara butiran $T_p$, pada suhu rendah yang lebih rendah apabila kepekatan Cd meningkat. Maka, ini boleh disimpulkan bahawa kesan magnet dinamik sampel bukan hanya yang dikira kebergantungan pada fasa tetapi ia juga bergantung kepada gandingan antara butiran. Nilai $I_0$ menunjukkan kualiti butiran gandingan dan nilai ini menurun apabila kepekatan cadmium bertambah.

Keputusan corak belauan XRD menunjukkan semua sampel yang didopkan dengan Cd, mengandungi puncak-puncak fasa 2212 dan ini menunjukkan kehadiran fasa rendah. Keamatan puncak-puncak bertambah apabila kepekatan Cd bertambah. Isipadu fasa...
2223 menurun berperingkat-peringkat mengikut peningkatan kepekatan Cd. Tambahan pula, ada kemungkinan ion Cd\(^{2+}\) menghuni tapak kekisi lain pada sampel.

Apabila masa persinteran dilanjutkan, sifat superkonduktor semakin jelas apabila didopkan dengan kepekatan yang rendah, \(x = 0.02\), di mana sampel ini masih didominasi oleh fasa 2223. Apabila kepekatan melebihi daripada 0.02, saiz butiran semakin mengecil, menjadi lebih pendek dan tebal, dengan taburan rawak jika dibandingkan dengan sampel. Ia juga menunjukkan bahawa sifat-sifat superkonduktor dan mikrostruktur semakin baik apabila sampel disinter selama 48 jam dan 100 jam, dengan fasa tinggi paling dominant dan membuktikan bahawa masa optimum persinteran mestilah lebih daripada 48 jam.

Kajian ini menunjukkan bahawa penggantian Cd pada tapak Ca tidak meningkatkan suhu genting, \(T_c\), bagi system BSCCO. Ini adalah disebabkan oleh pembentukkan fasa rendah yang melemahkan gandingan butiran. Walaubagaimana pun, semua sampel jelas menunjukkan peningkatan dalam \(T_c\) apabila masa persinteran meningkat.
ACKNOWLEDGEMENTS

In the name of Allah, the most Gracious and the most Merciful

Praise be to Allah the Almighty, for thee (alone) we worship and thee (alone) we ask for help. And praise be upon Mohammad s.a.w who his guidance has led us to the path whom God has favoured.

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To my father, my late mother, brothers and sisters, their love and support keep me going; and last but not least, to my wife, Aisha and my daughters, Fatema and Dwaa, thank you for your love, continuous support, encouragement and understanding.

May GOD Bless You All.
I certify that an Examination Committee met on 15/4/2004 to conduct the final examination of Ali Agail Hamed Abdulgader on his Master of Science thesis entitled “Effect of Cadmium Doping on BSCCO Superconductor Prepared via Coprecipitation Method” in accordance Universiti Pertanian Malaysia (Higher Degree) act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

____________________________________

ALI AGAIL HAMED ABDULGADER

Date:
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>DEDICATIONS</td>
<td>ii</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>ABSTRAK</td>
<td>vi</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>ix</td>
</tr>
<tr>
<td>APPROVAL</td>
<td>xi</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>xiii</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xvi</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>xvii</td>
</tr>
<tr>
<td>LIST OF PLATES</td>
<td>xxiii</td>
</tr>
<tr>
<td>LIST OF SYMBOLS AND ABBREVIATIONS</td>
<td>xxiv</td>
</tr>
</tbody>
</table>

## CHAPTER

### I INTRODUCTION

1. Basic Phenomena of the Superconductivity
   3
2. High $T_c$ Superconductors (HTS)
   5
3. Bi-Sr-Ca-Cu-O (BSCCO) System
   11
4. Application Of Superconductors
   13
   - Small-Scale Applications
   13
   - Large-Scale Applications
   14
5. Research Objectives
   16

### II LITERATURE REVIEW

1. Samples Preparation
   17
   - Coprecipitation Method
   18
   - Effect of Sintering Time
   20
2. Effect of Doping in BSCCO Superconductors
   24
   - Effect of Lead Doping
   25
   - Effect of Substitutional Doping in Ca Site
   28
   - Effect Of Substitutional Doping In Various Sites
   36

### III FUNDAMENTAL OF HTS AND MICROSCOPIC THEORY

1. Meissner – Ochsenfeld Effect
   41
   - Type I
   42
   - Type II
   44
2. Electronic Specific Heat
   45
3. Isotope Effect
   46
4. Microscopic Theory of Superconductors
   47
# IV PREPARATION AND CHARACTERIZATION TECHNIQUES

<table>
<thead>
<tr>
<th>Preparation Method</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Preparation</td>
<td>60</td>
</tr>
<tr>
<td>Heating Process</td>
<td>62</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Characterisation Technique</th>
<th>65</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistance Measurement</td>
<td>65</td>
</tr>
<tr>
<td>AC Magnetic Susceptibility Measure</td>
<td>66</td>
</tr>
<tr>
<td>X-ray Diffraction Analysis</td>
<td>69</td>
</tr>
<tr>
<td>Microstructure Analysis</td>
<td>70</td>
</tr>
</tbody>
</table>

# V RESULTS AND DISCUSSION

<table>
<thead>
<tr>
<th>Resistance Measurement</th>
<th>72</th>
</tr>
</thead>
<tbody>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>72</td>
</tr>
<tr>
<td>Bi$<em>{1.6}$Pb$</em>{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>72</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>79</td>
</tr>
<tr>
<td>Bi$<em>{1.7}$Pb$</em>{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>79</td>
</tr>
<tr>
<td>Ac Susceptibility Measurement</td>
<td>85</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>86</td>
</tr>
<tr>
<td>Bi$<em>{1.6}$Pb$</em>{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>86</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>109</td>
</tr>
<tr>
<td>Bi$<em>{1.7}$Pb$</em>{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>109</td>
</tr>
<tr>
<td>X-ray Diffraction Analysis</td>
<td>131</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>131</td>
</tr>
<tr>
<td>Bi$<em>{1.6}$Pb$</em>{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>131</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>137</td>
</tr>
<tr>
<td>Bi$<em>{1.7}$Pb$</em>{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>137</td>
</tr>
<tr>
<td>Microstructural Analysis</td>
<td>144</td>
</tr>
<tr>
<td>Bi$<em>{1.6}$Pb$</em>{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>144</td>
</tr>
<tr>
<td>Bi$<em>{1.7}$Pb$</em>{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>144</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>147</td>
</tr>
<tr>
<td>Bi$<em>{1.6}$Pb$</em>{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>147</td>
</tr>
<tr>
<td>Effect of Cd doping in Ca site of</td>
<td>150</td>
</tr>
<tr>
<td>Bi$<em>{1.7}$Pb$</em>{0.3}$Sr$_2$Ca$_2$Cu$_3$O$_8$</td>
<td>150</td>
</tr>
</tbody>
</table>

# VI CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

<table>
<thead>
<tr>
<th>Conclusions</th>
<th>151</th>
</tr>
</thead>
<tbody>
<tr>
<td>Suggestion for Future Work</td>
<td>154</td>
</tr>
</tbody>
</table>

# REFERENCES

155

# APPENDICES

163

# BIODATA OF THE AUTHOR

169